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Biocompatible Ionic Liquid Promote One-Pot Synthesis of 2-Amino-4H-Chromenes Under Ambient Conditions

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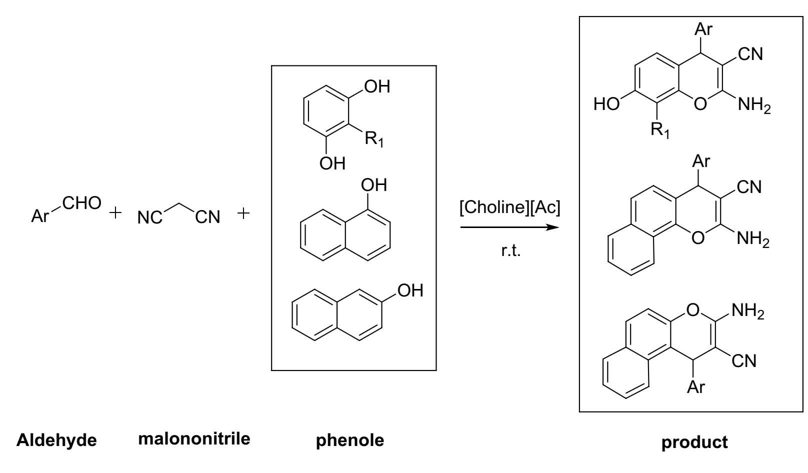
Abstract

The synthesis of 2-amino-4H-chromenes through one-pot multicomponent reactions has received great attention due to the high atom efficiency and the broad bioactivities of the products. Here, the catalytic performances of a series of functional ionic liquids towards this type of reaction were investigated and the results showed that the ionic liquid, choline acetate ([Choline][Ac]), could efficiently promote this reactions under room temperature without the necessary of organic solvents. The reaction is easy to be conducted and the following work-up procedures are very simple. The pure products can be obtained through extraction and washing procedures, no column separation procedures are needed. Intriguingly, this reaction system can be easily scaled up to multi-gram, suggesting its perspective in industrial applications. In addition, the ionic liquid [Choline][Ac] can be easily prepared from cheap and biocompatible materials, and it can be feasibly recycled and reutilized for at least five cycles.

Graphic Abstract

Various aldehydes, malononitrile and activated phenols could be converted to thecorresponding 2-amino-4H-chromenes with good to excellent isolated yields underthe catalysis of choline acetate at room temperature.



★ Cheap and biocompatible ionic liquid.

★Wide substrates generality.

★ Success in scale-up utilization.

★ Simple operation and work-up procedures.

★ Recyclable and reusable catalytic ionic liquid.

Keywords Ionic liquid · Choline acetate · 2-Amino-4H-chromenes · Room temperature

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Extended author information available on the last page of the article

1 Introduction

2-Amino-4H-chromenes and their derivatives have received great attention both in synthetic and biological aspects due to their wide range of antibacterial [1], anti-inflammatory [2], antioxidant [3], anti-hiv [4], and anti-cancer activities [5]. The most intriguing method for their preparation is through the one-pot multicomponent reactions(MCRs) using malononitrile, activated phenoles and various aldehydes [6]. The reported catalytic systems have been involved in tungstic acid functionalized mesoporous SBA-15 [7], mesolite [8], $Cu_{0.5}Co_{0.5}Fe_2O_4$ nanoparticles [9], nanosized MgO and Fe_3O_4 [10, 11], RGO-SO_3H [12], DABCO [13], {Mohr, 1975 #181}hydrotalcite [14], aqueous K_2CO_3 [15], Et_3N [16], $MeSO_3H$ [17], Sulfonic acid-functionalized MIL-101 [18], iodine [19], L-proline [20], $Fe(HSO_4)_3$ [21], DBU [22], Triethanolamine [23], molybdenum Schiff base [24], CNTs supported imidazolium-based ionic liquids [25, 26], and so on. Although these catalytic methods simplified the synthetic and work-up procedures with high atom economy coming from the inherent priorities of MCRs, most of them required the use of volatile organic solvents, high reaction temperature and suffered from the difficulties to reutilize the catalysts. In order to decrease the energy input, Dekamin et al. [27] found an mechanochemical ball milling method under the catalysis of potassium phthalimide at moderate temperature, but the catalyst was still difficult to be reused. So, the development of novel feasible and recyclable reaction systems for the synthesis of 2-amino-4H-chromenes with high catalytic activity under mild reaction conditions is still urgent.

Ionic liquids, as an excellent solvents and promising catalysts, have showed significant performances to improve the reactions through the modulating of reactivity and selectivity as well as facilitating the separation procedures, due to their easy accessibility and tunable physicochemical properties [28–30]. The ingenious utilization of the synergetic effects of cations and anions of ionic liquids has realized many harsh reactions with high efficiency under mild conditions, decreasing the consuming of organic solvents and the dependence of metal catalysts, and usually the whole time and energy input can also be cut down [31–34]. This in a great extent makes the whole related systems sustainable and accorded with the principles of green chemistry [35]. However, as it has been witnessed that although the ionic liquid catalytic systems can efficiently relief the utilization of organic solvents, their own synthetic procedures are usually tedious and the organic solvents are unavoidable [36–38], which without doubt will decrease the eco-efficiency of the whole production procedures. Therefore, the synthesis of ionic liquids from biocompatible and renewable resources with simple synthetic procedures is still in great needed for the further utilization of ionic liquids.

In our continuous work on the development and utilization of functional ionic liquids [32, 39–42], here, we found that an ionic liquid choline acetate ([Choline][Ac]) could promote the one-pot multicomponent reactions among aldehydes, malononitrile and activate phenols with excellent activity and selectivity for the target 2-amino-4H-chromenes under the solvent-free conditions at room temperature. No organic solvent was needed in this reaction system and after the reaction was completed, the products could be separated by simple extraction and wash procedures, no column separation procedures were necessary, which made the operation and work-up procedures very simple and applicable for industrial applications. The successful scale-up trial of this system to multi-grams and reutilization of ionic liquid to five cycles reinforced its industrial point. In addition, this ionic liquid could be prepared from biocompatible and cheap raw materials. So, the benefit from one-pot multicomponent reaction itself plus the convenient preparation and reutilization of ionic liquid, the concise operation and separation procedures made the reaction system developed in this work accorded greatly with the criteria of green chemistry.

2 Experimental

2.1 Chemical and Apparatus

Resorcinol, 2-methylresorcinol, *N*,*N*-dimethylethanolamine, diethanolamine, *N*,*N*-diethylethanolamine, betaine, 2-(methylamino)ethanol, triethanolamine, triethylamine, acetic acid (Ac), propionic acid (Pro), n-butyric acid (But), n-pentanoic acid (Pen), n-hexylic acid (Hex), lactic acid (Lac), malononitrile, benzaldehyde, 4-nitrobenzaldehyde, 3-nitrobenzaldehyde, 2-nitrobenzaldehyde, 2-furaldehyde, 4-fluorobenzaldehyde, 4-bromobenzaldehyde, 2-bromobenzaldehyde, p-methyl benzaldehyde, 4-methoxybenzaldehyde, 2-chlorobenzaldehyde, 3-chlorobenzaldehyde, 4-chlorobenzaldehyde with AR grade were purchased from Aladdin Reagents Company. DMSO-d_6 and choline hydroxide (45 wt% aqueous solution) were purchased from Sigma-Aldrich. Anhydrous ethanol and n-hexane were purchased from Sinopharm Chemical Reagent Co. The water used in this work was twice deionized.

^1H NMR spectra and ^{13}C NMR spectra were recorded on BRUKER AV-400 and BRUKER AV-600 instrument at room temperature by using TMS as internal standard. Melting points were taken from a XRC-1 Microscopic Melting Point Measurer (Sichuan University instrument factory) without correction.

2.2 Synthesis of Ionic Liquids

The ionic liquids were synthesized through the reactions of corresponding amines and acids. In a typical reaction for the synthesis of [DMEOA][Ac], 0.1 mol *N*,*N*-dimethylethanolamine (DMEOA) dissolved in 10 mL anhydrous ethanol was slowly added to equal molar acetic acid under stirring in an ice bath for 2 h, and then the mixture was reacted at 40 °C for further 24 h. The solvent was removed at 50 °C by rotary evaporation under reduced pressure and then dried under vacuum overnight to give the target ionic liquids. The other ionic liquids including [DEEOA][Ac], [DEOA][Ac], [TEOA][Ac], [Betain][Ac], [TEA][Ac], [MMEOA][Ac] were also synthesized according to the above procedures. Choline based ionic liquids like [Choline][Ac], [Choline][Pro], [Choline][Lac], [Choline][But], [Choline][Pen] and [Choline][Hex] were synthesized in a similar way from Choline hydroxide and corresponding acids.

2.3 Typical Reaction Procedure for the Synthesis of 2-Amino-4-H-Chromenes

Ionic liquid (0.5 mmol), phenols (0.5 mmol), aromatic aldehydes (0.5 mmol) were added to a 10 mL round-bottom flask successively and stirred magnetically, and then malononitrile (0.5 mmol) was added to the mixture at room temperature. The whole reaction process was monitored by TLC. After the reaction, the product was washed and centrifuged with secondary water to remove the ionic liquid, the obtained crude product was then recrystallized with ethanol to obtain the pure target product.

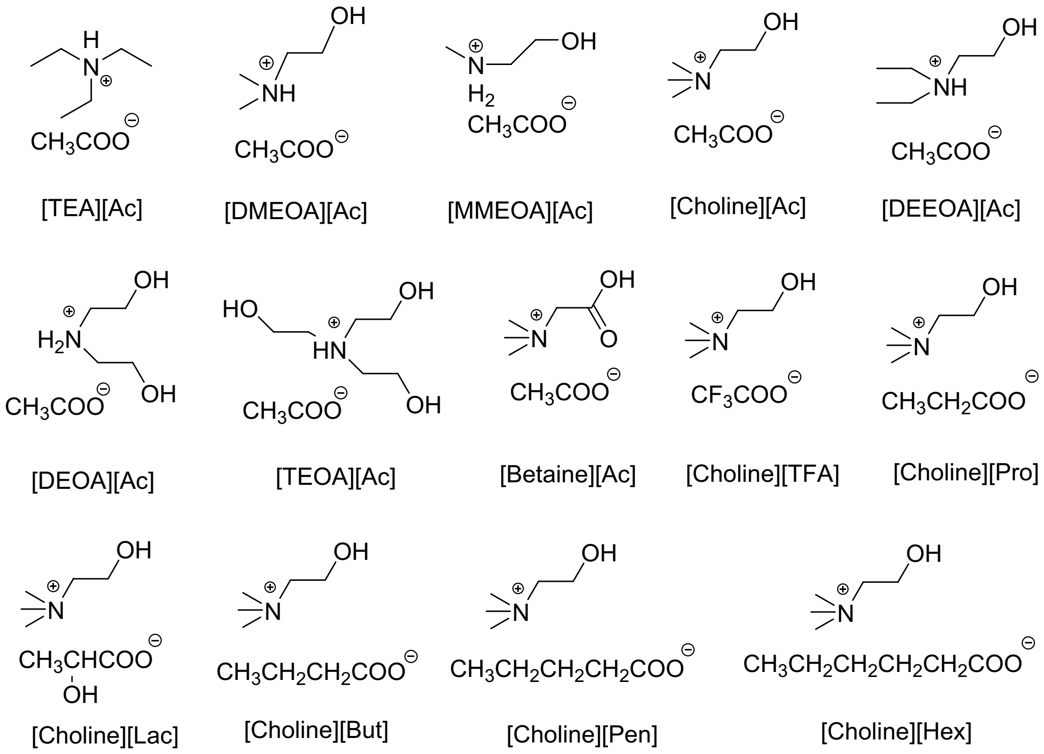
2.4 Recycle and Reutilization of Ionic Liquid

The ionic liquid extracted with water after the completion of reaction was rotary evaporated to remove most of the water and then it was dried in a vacuum drying oven overnight. The fully dried ionic liquid was directly used for the next reaction cycle.

3 Results and Discussion

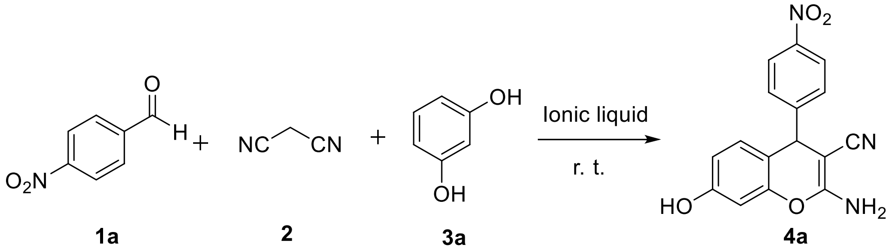
3.1 Structure Effect of Ionic Liquid on the Reaction

A series of ionic liquids based on ethanolamine were synthesized and illustrated in Scheme 1. Their performances in promoting the one-pot multicomponent synthesis of 2-amino-4H-chromenes were investigated using the reaction of p-nitrobenzaldehyde, malonitrile and resorcinol as a model and the results were collected in Table 1.The results showed that when acetate anion was used, the ionic liquids



Scheme 1 The structures of ionic liquids

Table 1 The effect of different ionic liquids on the reaction



Entry[a]	IL	Yield (%)[b]	Entry[a]	IL	Yield (%)[b]
1	[TEA][Ac]	50	8	[Betain][Ac]	10
2	[DMEOA][Ac]	90	9	[Choline][TFA]	8
3	[MMEOA][Ac]	89	10	[Choline][Pro]	87
4	[Choline][Ac]	96	11	[Choline][Lac]	60
5	[DEEOA][Ac]	60	12	[Choline][But]	80
6	[DEOA][Ac]	72	13	[Choline][Pen]	78
7	[TEOA][Ac]	70	14	[Choline][Hex]	85

[a] Reaction conditions: resorcinol (0.5 mmol, 55.1 mg), p-nitrobenzaldehyde (0.5 mmol, 75.6 mg), malononitrile (0.5 mmol, 33.0 mg), IL (0.5 mmol, 1.0 eq), room temperature, 3 h

[b] Isolated yield

with single hydroxyethly group on the nitrogen atom of cations like MMEOA, DMEOA, and cholinum have excellent performance towards the target products (entries 2–4, Table 1), while ionic liquids with two ethyl groups or two or three hydroxyethyl groups like DEEOA, DEOA and TEOA only have moderate promoting effect (entries 6–7, Table 1). However, the ionic liquids without hydroxyethyl group or with carboxyl group on the cation like TEA or betaine have poor catalytic activity (entries 1, 8, Table 1).The anion effect of ionic liquids was investigated combining with the choline cation. The results showed that the acetate anion has the most excellent performance (entries 4, 9–14, Table 1), the target product can arrive at 96% isolated yield under room temperature. Although the further increase of the reaction temperature to 40 °C using [Choline][Ac] as catalyst can lead the completion of the reaction within 1 h, the room temperature was still selected for the convenient operation and lower energy input. The excellent performance of [Choline][Ac] comparing with the other ionic liquids suggests the importance of the synergetic effect of the cation and the anion of ionic liquids. The proper basicity coming from the anion and proper hydrogen bond donor ability from cation of the ionic liquids are essential for the catalytic procedures.

3.2 Amount Effect of Ionic Liquid on the Reaction

According to the well-deserved performance of the optimized ionic liquid [Choline][Ac] in the production of 2-amino-4H-chromenes, its amount effect on the model reaction was investigated and the results were given in Table 2. The results showed that the yield for the target compound increased with the increase of the ionic liquid molar ratio to substrates from 0.01 to 1.0 (entries 1–7, Table 2). When the ionic liquid dosage was 1.0 equivalent, the yield reached the highest, and the further increase of the ionic liquid amount

Table 2 The amount effect of ionic liquid on the model reaction

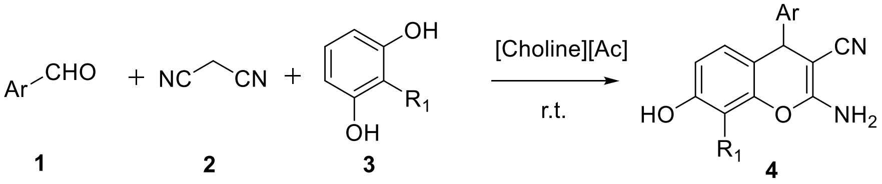
Entry[a]	Ratio[b]	Yield (%)[c]
1	0.01	53
2	0.02	55
3	0.05	58
4	0.1	85
5	0.2	88
6	0.5	90
7	1.0	96
8	1.5	94

[a] Reaction conditions: resorcinol (0.5 mmol, 55.1 mg), p-nitrobenzaldehyde (0.5 mmol, 75.6 mg), malononitrile (0.5 mmol, 33.0 mg), [Choline][Ac], room temperature, 3 h

[b] The molar ratio of ionic liquid to substrates

[c] Isolated yield

Table 3 Synthesis of various 2-amino-4H-chromene derivatives from aldehydes, malonitrile and resorcinols



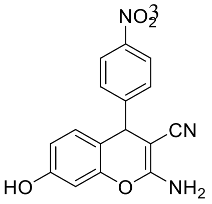
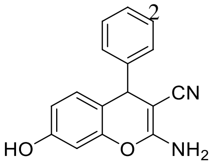
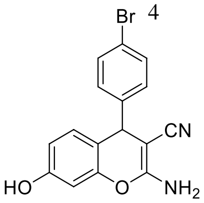
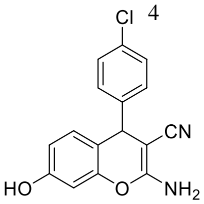
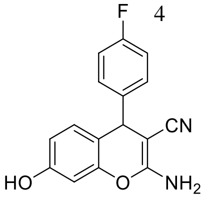
Entry[a]	Ar	R_1	Product	Time (h)	Yield (%)[b]	M.P. (°C)	
						Found	Report(Lit)
1	$4\text{-}NO_2C_6H_4$	H	 **4a**	3	96	161–163	166–168 [27]
2	C_6H_5	H	 **4b**	2	70	226–228	235–237 [27]
3	$4\text{-}BrC_6H_4$	H	 **4c**	4	80	220–222	222–224 [27]
4	$4\text{-}ClC_6H_4$	H	 **4d**	4	80	160–162	161–163 [27]
5	$4\text{-}FC_6H_4$	H	 **4e**	4	85	216–218	191–192 [27]

Table 3 (continued)

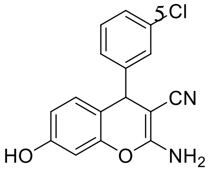
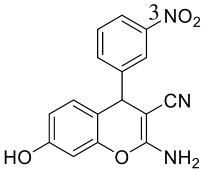
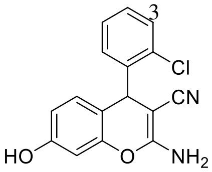
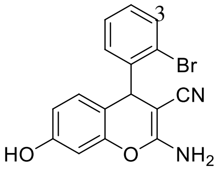
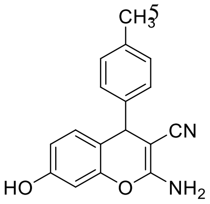
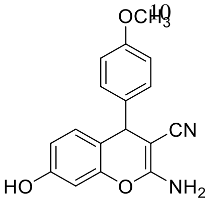
Entry[a]	Ar	R_1	Product	Time (h)	Yield (%)[b]	M.P. (°C)	
						Found	Report(Lit)
6	3-ClC_6H_4	H	 **4f**		90	104–106	106–109 [43]
7	3-$NO_2C_6H_4$	H	 **4g**		90	174–176	167–169 [27]
8	2-ClC_6H_4	H	 **4h**		82	100–101	97–99 [27]
9	2-BrC_6H_4	H	 **4i**		90	206–208	205–206 [12]
10	4-$CH_3C_6H_4$	H	 **4j**		85	196–198	188–190 [27]
11	4-$OCH_3C_6H_4$	H	 **4k**		88	118–120	110–112 [27]

Table 3 (continued)

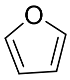
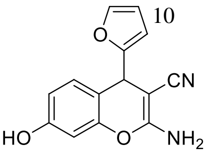
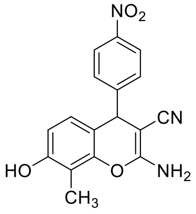
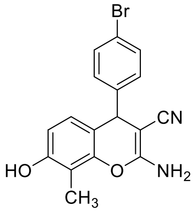
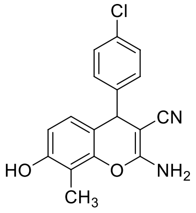
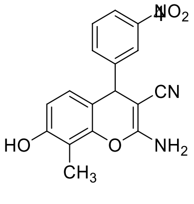
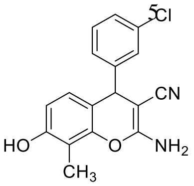
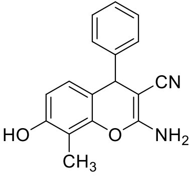
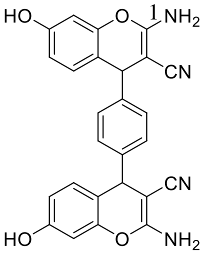
Entry[a]	Ar	R_1	Product	Time (h)	Yield (%)[b]	M.P. (°C)	
						Found	Report(Lit)
12		H	 **4l**	10	70	216–218	190–192 [27]
13	$4\text{-}NO_2C_6H_4$	$2\text{-}CH_3$	 **4m**	4	92	110–112	–
14	$4\text{-}BrC_6H_4$	$2\text{-}CH_3$	 **4n**	12	60	128–130	–
15	$4\text{-}ClC_6H_4$	$2\text{-}CH_3$	 **4o**	9	70	122–124	220–221 [44]
16	$3\text{-}NO_2C_6H_4$	$2\text{-}CH_3$	 **4p**	4	92	108–110	–

Table 3 (continued)

Entry[a]	Ar	R_1	Product	Time (h)	Yield (%)[b]	M.P. (°C)	
						Found	Report(Lit)
17	$3\text{-}ClC_6H_4$	$2\text{-}CH_3$	 **4q**	5	86	148–150	–
18	C_6H_5	$2\text{-}CH_3$	 **4r**	12	77	128–130	–
19	$OHCC_6H_4$	H	 **4s**	1	81	> 300	> 300 [11]

[a]Reaction conditions: resorcinol (0.5 mmol, 55.1 mg), aromatic aldehydes (0.5 mmol), malononitrile (0.5 mmol, 33.0 mg), [Choline][Ac] (0.5 mmol, 81.5 mg)

[b]Isolated yield

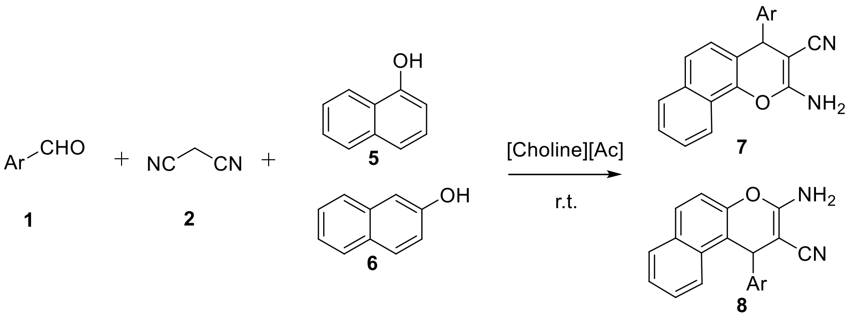
has no significant influence on the target compound production (entry 8, Table 2).

3.3 [Choline][Ac] Catalyzed Synthesis of 2-Amino-4H-Chromenes

The generality of this reaction system was firstly investigated using the reactions of various aromatic aldehydes, malonitrile and resorcinols. The results collected in Table 3 showed that different aromatic aldehydes with various substitutes like electron-donating or electron-withdrawing groups, or halogen groups could react with malonitrile and resorcinol smoothly, leading to the satisfactory isolated yields for the target yields within adequate reaction time (Entries 1–11, Table 3). The heterocyclic aromatic aldehyde (Entry 12, Table 3) could also arrive at good isolated yields after 10 h reaction. The reaction rate of 2-methylresorcinolwas a little sluggish but it could also reacted with various aldehydes and malonitrile, giving the target compounds with satisfactory yields with a little prolonged reaction time(Entries 13–18, Table 3). An interesting results was also obtained for the reaction of terephthalaldehyde, two of its aldehyde groups could both reacted with resorcinol and malonitrile with exclusive chemo-selectivity (Entry 19, Table 3).

Furthermore, the reactivity of other activated phenols like 1-naphthol and 2-naphthol with aldehydes and malononitrile were also investigated to demonstrate the generality of this novel reaction system. The results collected in Table 4 showed that the reactivity of 1-naphthol was higher than that

Table 4 Synthesis of various 2-amino-4H-chromene derivatives from aldehydes, malonitrile and naphthols



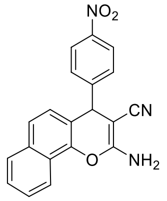
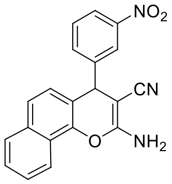
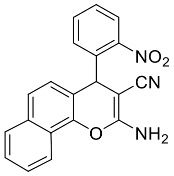
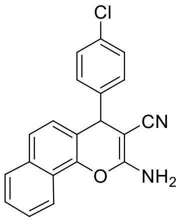
Entry[a]	Ar	Product	Time (h)	Yield (%)[b]	M.P. (°C)	
					Found	Report(Lit)
1	4-$NO_2C_6H_4$	 **7a**	0.5	95	214–215	233–234 [27]
2	3-$NO_2C_6H_4$	 **7b**	0.6	94	206–208	211–213 [27]
3	2-$NO_2C_6H_4$	 **7c**	1	94	214–216	221–224 [24]
4	4-ClC_6H_4	 **7d**	2	86	239–240	232–234 [27]

Table 4 (continued)

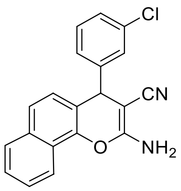
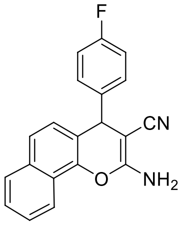
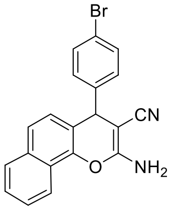
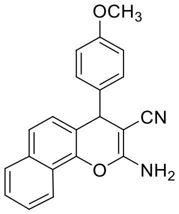
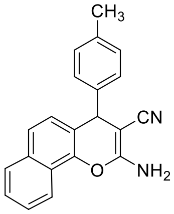
Entry[a]	Ar	Product	Time (h)	Yield (%)[b]	M.P. (°C)	
					Found	Report(Lit)
5	3-ClC_6H_4	 **7e**	5	82	205–207	212–214 [23]
6	4-FC_6H_4	 **7f**	4	80	215–216	233–235 [27]
7	4-BrC_6H_4	 **7g**	2	86	240–242	232–234 [27]
8	4-$OCH_3C_6H_4$	 **7h**	7	82	139–140	187–189 [27]
9	4-$CH_3C_6H_4$	 **7i**	7	65	177–179	206–208 [27]

Table 4 (continued)

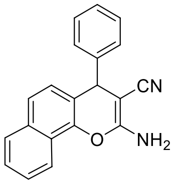
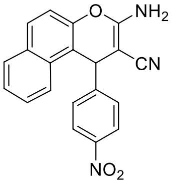
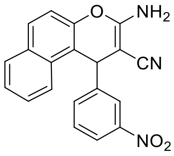
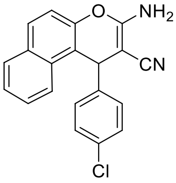
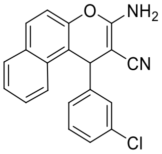
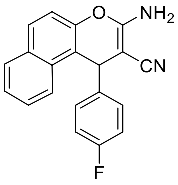
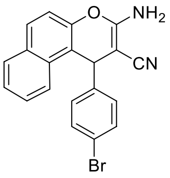
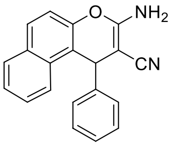
Entry[a]	Ar	Product	Time (h)	Yield (%)[b]	M.P. (°C)	
					Found	Report(Lit)
10	C_6H_5	 **7j**	4	80	138–140	178–180 [10]
11	4-$NO_2C_6H_4$	 **8a**	7	95	142–144	187–189 [27]
12	3-$NO_2C_6H_4$	 **8b**	7	85	228–230	232–234 [27]
13	4-ClC_6H_4	 **8c**	10	75	154–156	173–175 [26]
14	3-ClC_6H_4	 **8d**	10	89	234–236	234–236 [45]

Table 4 (continued)

Entry[a]	Ar	Product	Time (h)	Yield (%)[b]	M.P. (°C)	
					Found	Report(Lit)
15	4-FC_6H_4	 **8e**	14	90	222–224	226–228 [45]
16	4-BrC_6H_4	 **8f**	14	87	210–212	238–240 [27]
17	C_6H_5	 **8g**	10	75	260–262	283–285 [27]

[a] Reaction conditions: naphthol (0.5 mmol, 72.1 mg), aromatic aldehydes (0.5 mmol), malononitrile (0.5 mmol, 33.0 mg), [Choline][Ac] (0.5 mmol, 81.5 mg)

[b] Isolated yield

of 2-naphthol, and both of them could be converted to the target products with satisfactory yields.

3.4 Scale-up Experiment

Considering the good generality of this novel reaction system in various aldehydes and phenols, we explored the scale-up experiment to investigate its potential utilization in industrial production. Ionic liquid (10 mmol), resorcinol (10 mmol), p-nitrobenzaldehyde (10 mmol) and malononitrile were added to a 25 mL round-bottom flask in turn and stirred for 3 h at room temperature. The whole reaction process was monitored by TLC. After the purification procedures, the pure product can arrive at 97%. After the reaction was completed, 2 ml water was added to the reaction mixtures, the crude product could be precipitated. The pure product was then obtained by simple washing procedures and the ionic liquid could be reutilized after simple drying.

3.5 Reusability of the Ionic Liquid

The reutilization of ionic liquid on the model reaction was investigated and the results were illustrated in Fig. 1. It was showed that the catalytic activity of choline acetate have no significant decrease after five cycles, suggesting the excellent reusability. The FT-IR spectra of the fresh and the reused ionic liquid have also been investigated and the results were shown in Fig. 2. The results showed that this ionic liquid have good stability in the reaction and recovered procedures.

3.6 Possible Mechanism

In the reaction procedure, the Knoevenagel reaction product was monitored to be the reaction intermediate. According to our previous work about the utilization of hydrogen bond functionalized ionic liquids in synthesis, it was deduced that

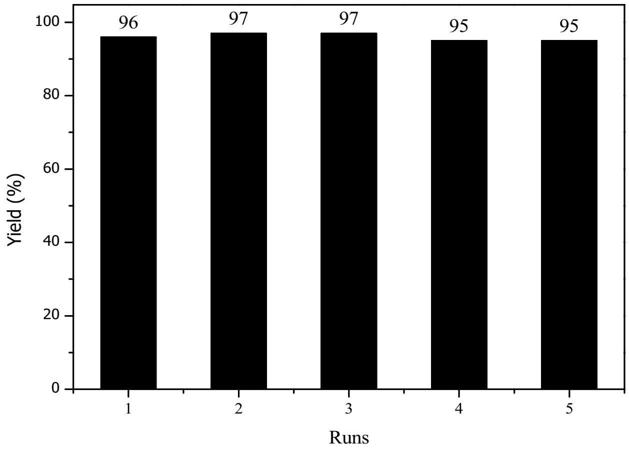


Fig. 1 The reutilization of ionic liquid

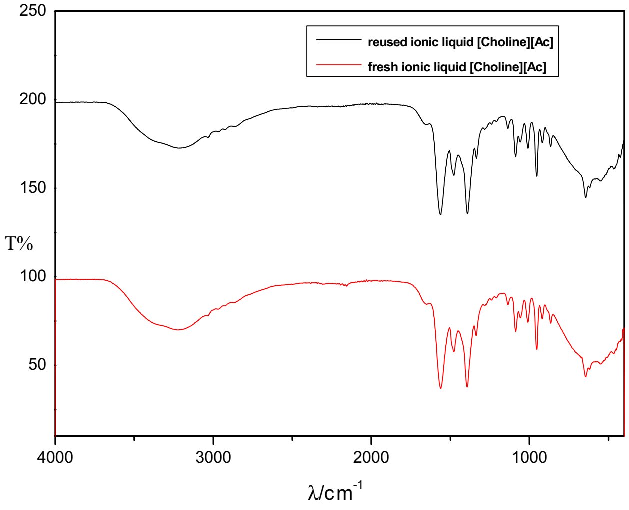


Fig. 2 FT-IR for the fresh and reused ionic liquid

the hydroxyl group on the ionic liquid cation could activate the C=O of aldehyde, the ionic liquid anion actived the α-H of malonitrile, leading to the formation of carbon anions. Then the carbon anion attacked the carbonyl groups of aldehyde to produce intermediate I. On the other hand, the resorcinol was dehydrogenated by ionic liquid to produce Phenoxy anion, which then reacted with intermediate I, and after the following cyclization rearrangement procedures to produce the target products (Scheme 2).

4 Conclusions

In this work, an efficient ionic liquid based catalytic system for the preparation of 2-amino-4H-chromenewas was reported. This ionic liquid could be easily prepared from cheap and biocompatible materials. The reaction system could be conducted at room temperature without the necessary of additional organic solvents, and the work-up procedures were very simple, no column purification was needed. The following experiment suggested that the reaction system could be scaled up to multi-gram scale. In addition, this ionic liquid could be convenient recycled and reutilized for at least five cycles without significant activity decrease. The above benefits of this reaction system showed that it is promising for the further utilization in industrial applications.

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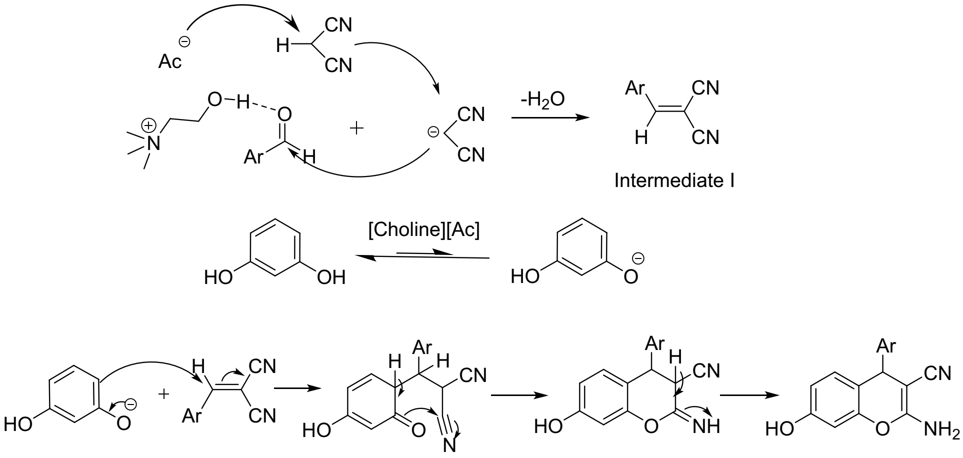
Scheme 2 The plausible reaction mechanism



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